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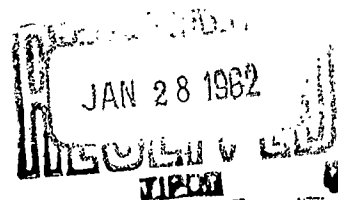
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ELECTRON MICROSCOPIC FRACTOGRAPHY

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ELECTRON MICROSCOPIC FRACTOGRAPHY

W. R. Warke and A. R. Elsea*

INTRODUCTION

In the past few years, a valuable metallurgical tool has been developed which can be used in solving both theoretical and applied metallurgical problems. This tool is known by various names including electron microscopic fractography, electron fractography, electron microfractography, submicrotopography or simply fractography. As the names imply, it consists of examining fracture surfaces (actually replicas of fracture surfaces) under the electron microscope. Significant information regarding the micromechanism of various types of fracture has been obtained using this technique. Furthermore, fractures produced by different mechanisms or in different ways have characteristic appearances when viewed under the electron microscope. Therefore, electron microfractography is becoming a very valuable tool for use in service failure analysis.

Fractography, the examination at high magnification of fracture surfaces, is a term coined a number of years ago by Zapffe.⁽¹⁾ He and his co-workers pioneered in this field, using the light microscope at magnifications up to about 1000X. Fractography with the light microscope was extremely tedious and time-consuming because of the limited depth of focus at high magnification and the hours required to get a facet of the fracture normal to the light beam. As a result, light microscopic fractography did not attract much attention and became nearly dormant.

Because of its greater depth of focus the electron microscope is more suited for fractography than is the light microscope. Thus, since the electron microscope has become a rather common instrument in modern metallurgical laboratories, interest in fractography has revived. Although some fractographic work is still done with the light microscope, much of the effort has involved the use of the electron microscope and electron microscopic fractography has become a highly reliable tool in the hands of those experienced in its use. This memorandum defines those areas of application where electron microscopic fractography is being used, and also those areas where limitations and lack of knowledge exist, thus should help stimulate further advances in this field.

SUMMARY

Electron microscopic fractography is the examination of fracture surfaces at relatively high magnification using the electron microscope. In order to do this, a replica of the fracture must be prepared. The two most

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common techniques of replication for fractography are the single stage, direct carbon method and the two stage, cellulose acetate-carbon method. The former technique gives a truer representation of the fracture surface but requires the destruction of the underlying metal in the course of replication. Therefore, direct carbon replication is generally used for research investigations where loss of the metal specimen may be of no concern. Plastic-carbon replication is used in service-failure analysis where the sample must be retained for other studies. Artifacts occur during replication and must be either recognized as such or eliminated so that they will not influence the interpretations. There is, at the present time, a controversy among the various investigators as to whether certain features are or are not artifacts.

A ductile, overload fracture has a characteristic appearance under the electron microscope, consisting of roughly elliptical domains which have been termed ductile dimples. It is thought that the fracture occurs by the formation, growth, and union of internal ruptures at inclusions. Each internal rupture produces a dimple on the fracture surface. From this, it can be deduced that ductility (i.e., per cent elongation) is directly related to the nature, amount, and distribution of the inclusions in the metal.

Fatigue fractures, at least in lower strength metals, are characterized by closely spaced striations on the fracture surface. Each stress cycle is believed to produce one striation on the fracture, and mechanisms by which the striations form have been proposed. The spacing of the striations is a function of the material, the stress cycle, and the environment, among other things, and some results of a quantitative nature have been obtained in this regard. Unfortunately, fatigue fractures in very high-strength steels do not show striations. Therefore, although the fracture appearance may be sufficiently characteristic to identify fatigue as the mechanism of fracture propagation, with the present state of the art the factors which influence fracture appearance cannot be evaluated quantitatively.

Intergranular fractures can be produced or promoted by a number of mechanisms such as hydrogen-induced failure, stress-corrosion cracking, quench cracking, and intergranular precipitation. At the present time, it is not possible to differentiate among all of the different types of intergranular fracture. It is hoped that further research will uncover characteristic features which will allow the various types of intergranular fracture to be distinguished. Grain boundary precipitates such as aluminum nitride for alloy steel and chromium carbide in stainless steel, which, in the presence of appropriate stress fields cause intergranular fractures have been identified and analyzed by diffraction techniques, using extraction replicas of the fracture surface.

Very few systematic investigations have been made to study the effects of composition, strength level, stress level, strain rate or other material and loading variables on the three types of failure mentioned above (overstress, fatigue, and intergranular). It is hoped that research programs of this type will be undertaken in the near future to increase our knowledge of the mechanisms of fracture as well as to increase capabilities for service-failure analysis.

TECHNIQUES, LIMITATIONS, AND ARTIFACTS

The techniques of replication and examination of fracture surfaces for electron microscopic fractography are essentially the same as those used on polished and etched specimens for electron metallography. As yet, no new techniques have been developed specifically for fractographic examination, although, perhaps, some could be used.

To begin with, it is of prime importance that a clean fracture surface be obtained. This is generally quite easy in research investigations where fractures are produced in the laboratory under closely controlled conditions. On the other hand, it is difficult to obtain a service failure in which the fracture surface has been protected from dirt, rusting, and abrasion before, during, and after the part has been removed from service. More care in handling parts that have failed in service is necessary when the fracture is to be examined by microfractography rather than visually at low magnifications. Furthermore, care must be exercised when cleaning the fracture with solvents, ultrasonic baths, or repeated stripping of plastic replicas to prevent further damage to the characteristic features of the fracture face.

Two standard methods of replication are used for electron microfractography. These are the two stage plastic-carbon technique and the single stage direct-carbon technique. Plastic-carbon replication consists, first of all, of obtaining a good plastic negative replica of the fracture surface. One of the more popular methods of doing this is to press a piece of cellulose-acetate sheet, which has been softened on one side with acetone, against the fractured surface. This plastic strip is then allowed to harden in place, producing a negative replica of the surface. The hardening process is thought to occur, for the most part, by the diffusion of acetone from the wet interface region into the dry portions of the plastic sheet, remote from the interface. If an excessive amount of acetone is used, it may evaporate at the interface producing an artifact in the form of small bubbles in the plastic replica.

After the plastic is allowed to harden it is stripped from the fracture and placed in a vacuum evaporator. In the evaporator, a shadowing material is deposited, if such is desired, and a layer of carbon is deposited. More will be said about shadowing later in this memorandum. The plastic-shadow-carbon composite is then treated in a manner so as to dissolve the plastic. This is done by immersion in acetone, which may be either at room temperature or warmed slightly to produce a higher rate of solution, or by placing the composite in vapors of acetone. Some difficulty is experienced at this stage in the processing due to the fact that the cellulose-acetate expands to approximately one and a half times its original volume during solution in acetone. This produces cracks and distortion in the carbon, which may interfere with examination of the replica or, in severe cases, may render it useless. A number of techniques are used to minimize the cracking problem. These include the use of very thin cellulose acetate sheet or small pieces cut from a larger replica, warming of the acetone used for solution of the plastic, or the embedding of a grid in the plastic to reinforce it and

restrict expansion during solution. This last technique is used by the Naval Research Laboratory and is accomplished by interposing a grid between the plastic and the specimen prior to replication and forcing the softened plastic through the grid openings, onto the fracture surface.

The freed carbon replica is dried, placed on a grid, and examined in the electron microscope.

Direct carbon replication consists of depositing the shadowing material and a layer of carbon directly on the fracture surface. The deposition is carried out in a vacuum evaporator in the same manner as that used for plastic-carbon replicas. The replica is removed from the specimen by etching (for example, nital or 10 per cent bromine solution can be used for steel), or electropolishing away the surface of the specimen, under the carbon layer. The carbon replica, which is thus freed, is subsequently cleaned in acid baths, rinsed and dried. The replica is then ready for examination in the microscope.

Each of these two replication techniques has its limitations and its areas of maximum applicability. The plastic-carbon technique is quite rapid and is nondestructive, the fracture surface being preserved for further study if desired. However, plastic-carbon replicas are more susceptible to artifacts than are the direct-carbon replicas. The direct carbon method is also rapid and gives the maximum obtainable fidelity of reproduction of the surface, but as it is presently used, requires etching of the surface to free the replica. Therefore, plastic-carbon replication is used, for the most part, for service failure analysis, where preservation of the specimen is of prime importance, and the direct-carbon technique is used mainly for research work on laboratory produced fractures where maximum fidelity is of prime importance.

An interesting variation of the direct carbon replication technique is the carbon-extraction replica method. Quite often, precipitate particles on the fracture surface will be extracted by a normal carbon replica^(2,3) but if the surface is very lightly etched prior to deposition of carbon, much additional information is obtained. Not only are more precipitates extracted in the replica, but the information regarding the relationship of the fracture to the underlying structure is obtained.⁽⁴⁾ Unknown precipitates can then be analyzed by X-ray or electron diffraction of the particles extracted on the replica. Several types of fracture are directly related to inclusions and precipitates. The nature of the fracture process will cause the particles to be concentrated on the fracture surface, where they are readily picked up and analyzed by the above technique.

Shadowing, although it is not considered to be critical in fractographic work, is one point on which there is a good deal of controversy among the workers in the field. The following tabulation lists a number of companies who have done fractographic work and the type of shadowing technique each has used.

<u>Company</u>	<u>Plastic-Carbon</u>	<u>Direct Carbon</u>
Douglas Aircraft Company	Chromium	Carbon
Boeing Company	Germanium	Germanium
Naval Research Laboratory	Palladium	Palladium
Crucible Steel Company	Uranium	--
U. S. Steel Corporation	Chromium	--
General Electric	Chromium	Carbon
Royal Aircraft Establishment	Gold-palladium	Gold-palladium
IRSID	--	None
Battelle Memorial Institute	Platinum-carbon	--

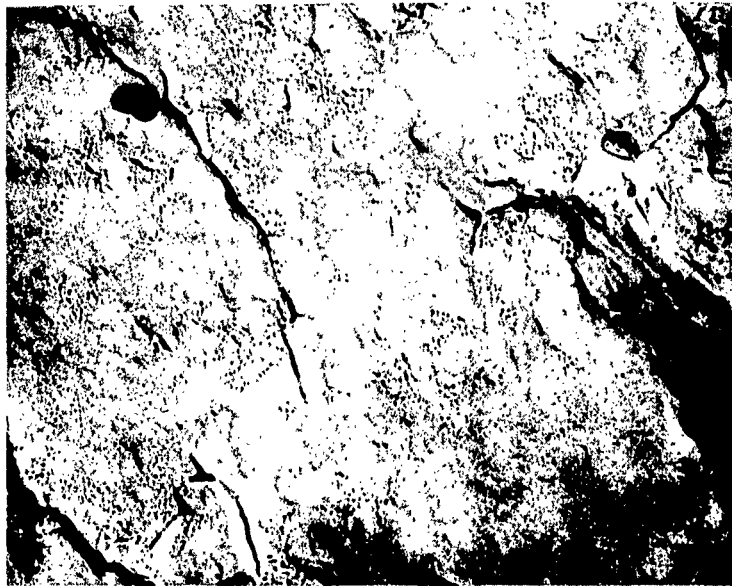
As can be seen, chromium is the most commonly used shadowing material. The shadow material must meet certain requirements. It must deposit uniformly, and it must not deteriorate in the electron beam. However, the features of most fractures are sufficiently coarse to be distinguished without the aid of shadowing, and some investigators, such as Plateau of IRSID, felt that shadowing only complicates and renders interpretation more difficult. The use of shadowing on certain flat, featureless types of fracture holds some merit, though, and if the macroscopic direction of crack propagation is known, shadowing can be used to maintain orientation of the replica with respect to this direction. But, the shadowing material seems to be of minor importance and an initial layer of carbon, deposited at an angle, followed by normal deposition may suffice.

The replica is examined in the electron microscope in the normal manner. Relatively low magnification is used, from 1500 to 10,000 diameters being the usual range. Higher magnifications are sometimes obtained when the fractographs are enlarged during printing.

Stereo viewing is used by several investigators and is felt, by them, to be extremely valuable. Stereo pairs are obtained by taking two fractographs of the same area on a replica with the replica at slightly different angles to the electron beam. The three dimensional effect thus obtained gives a much better indication of the coarseness, and the relative elevation of various aspects of the fracture.

The various artifacts obtained in replicas for electron microfractography are the cause of considerable controversy among the various investigators. Some of the workers in this field are interpreting as significant features of the fractures those markings that other workers consider to be mere artifacts in the replicas. As mentioned above, plastic-carbon replication is more susceptible to artifacts than direct carbon, and in order to ascertain what is or is not an artifact fractographs obtained using both techniques on the same fractures should be compared. Some examples of such controversial artifacts are hydrogen-indication sites, glide-plane decohesion striations, and flaps of plastic.

Hydrogen-indication sites were noted by Phillips and Bennett⁽⁵⁾ on a fracture produced in AISI 4340 by cathodic charging with hydrogen followed by application of a static load. Figure 1, which is taken from their paper, is the microfractograph of this fracture which was made using a chromium shadowed two-stage replica. The hydrogen-indication sites appear as small voids distributed on what may be subgrain boundaries. Ryder⁽⁶⁾,



27,000X

FIGURE 1. DELAYED FAILURE IN HYDROGEN EMBRITTLED AISI 4340 SHOWING VOIDS AT SUBGRAIN BOUNDARIES WHICH ARE CLAIMED TO BE HYDROGEN-INDICATION SITES⁽⁵⁾

of the Royal Aircraft Establishment in a forthcoming discussion of the above paper states that he, too, has seen such voids. However, he claims that the small voids are artifacts of the two-stage replication technique and are not found in bromine-stripped carbon replicas of fracture surfaces mating with those on which they are found using cellulose acetate. He is uncertain as to the source of the voids and feels that for high resolution work, bromine-stripped direct carbon replicas are superior. Beachem⁽⁷⁾ of NRL, has noticed similar voids in cellulose acetate-carbon replicas and connected their occurrence with the use of an excessive amount of acetone in softening the plastic sheet. He theorizes that the acetone evaporates at the metal-plastic interface while the plastic is still soft, thus producing a small round void in the plastic.

Another common feature, which is sometimes claimed to be significant, but which may be an artifact, are the striations observed in ductile fractures which are taken as being evidence of glide-plane decohesion. Glide-plane decohesion is a term which was coined by Crussard and his co-workers at IRSID⁽⁸⁾ to describe those areas on a ductile fracture which do not have the typical dimpled appearance. Figure 2 is an electron microfractograph from their paper which was made from a direct carbon replica of a ductile fracture in mild steel. Figure 3 is a fractograph showing striations in a brittle, intergranular fracture.⁽⁹⁾ These striations are taken as evidence of a region of ductile fracture by decohesion on a glide plane where slip has occurred.⁽⁴⁾ However, other investigators claim that these striations are not the result of glide-plane decohesions as described by Crussard, but are an artifact produced when the replica is scraped across a ledge on the fracture surface during stripping and that they can be avoided by more careful stripping or by the use of direct carbon replicas. A similar possible source of the striations is the dragging of the two faces of the fracture across one another in the course of separation.

Another artifact which leads to confusion is one which is sometimes termed "flaps of plastic" or folds. These appear as black areas on the fractographs, and typical examples can be seen in most of the fractographs in this report. Some investigators feel that these black areas are merely artifacts of the replication method while others claim that they represent sudden changes in elevation on the fracture surface⁽¹⁰⁾.

The cracks in the positive carbon replicas produced by expansion of the cellulose acetate during solution in acetone are an artifact which is minimized by careful technique and generally is ignored by all of the present workers in fractography.

DUCTILE FRACTURE

As a result of electron microfractographic studies^(8,11) and metallographic investigations^(11,12), an acceptable theory of the mechanism of ductile fracture has been obtained⁽¹²⁾. The fracture occurs first of all, by the formation of internal voids at the interface between the matrix and incoherent precipitates and inclusions or by the fracture of precipitate

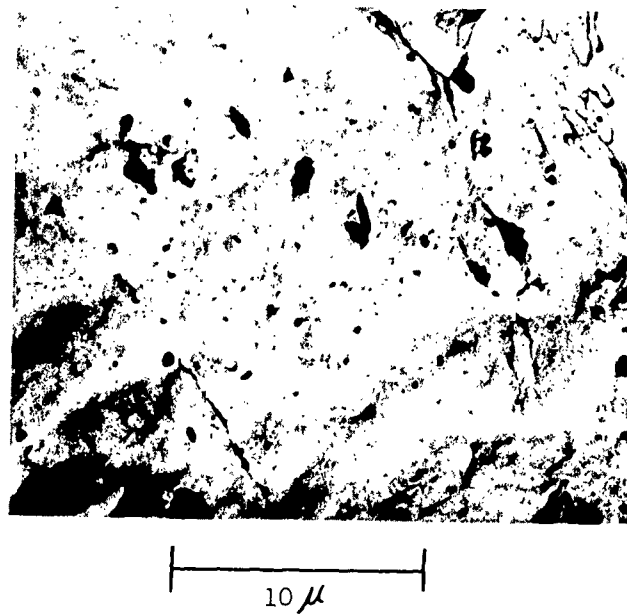


FIGURE 2. TYPICAL MICROFRACTOGRAPH OF GLIDE-PLANE DECOHESION IN MILD STEEL(8)



6,200X

FIGURE 3. "GLIDE-PLANE DECOHESION STRIATIONS" TAKEN TO INDICATE A DUCTILE REGION IN AN OTHERWISE BRITTLE INTERGRANULAR FRACTURE(9)

particles. Next, these voids grow as the metal elongates until finally rupture occurs by internal necking of the material between the voids. Each void subsequently appears on the fracture surface as a cuplike depression or dimple. A dimpled appearance, then, is characteristic of electron microfractographs of ductile failures.

Figure 4 is a series of microfractographs of a variety of materials including high strength steel, stainless steel, a nickel-iron alloy, and OFHC copper(8,11,13). These fractographs indicate that the dimpled appearance is typical of ductile failures in all metals; it is not restricted to any particular type of metal. Also, since a wide range of strength levels is represented, the same mechanism is evidently active and the same appearance results without regard to hardness and strength level.

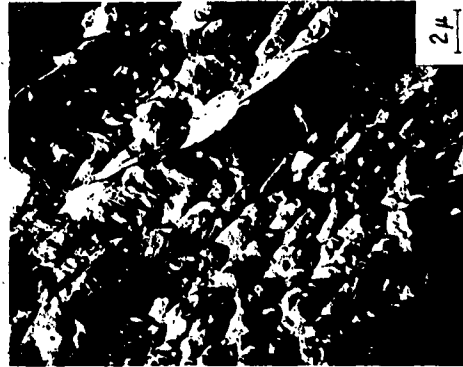
Figure 5 is a comparison of tensile and shear fractures in AISI 4340 steel(5,9). It will be noted that in the tensile fracture, the dimples are slightly elongated in random directions. This type of appearance is found in the flat transverse portion of tensile fractures. On the other hand, the dimples on the shear fracture are highly elongated in the direction of relative motion of the two fracture faces. This type of appearance is found in shear failures and in the shear-lip portion of tensile failures. It was thought at one time(5) that the direction of elongation of the ductile dimples could be used to ascertain the direction of crack propagation. It is now generally agreed that this is not so, but rather when shearing has occurred, the dimples point in opposite directions on opposite faces, as mentioned above, and when tearing has occurred the dimples point in the same direction on both faces. Another feature of the shear fracture is the appearance of a greater amount of "glide-plane decohesion".(8) Glide-plane decohesion is evidenced by relatively flat featureless areas on ductile fractures and is thought to occur by separation along planes that have been weakened by slip.

As was pointed out above, each void which appears as a dimple on the fracture surface, is nucleated at an inclusion. In Figures 4 and 5, many inclusions can be seen associated with the dimples. The relative size of the dimples is related directly to the size, nature, and distribution of the inclusions and precipitates in the material and is independent of the strength, composition or other material or loading variables. A fine, dispersed precipitate will produce small dimples and coarse precipitates will produce large dimples, so the dimple-size distribution will reflect the precipitate-size distribution. In fact, a material with particles of two distinct sizes (i.e., undissolved intermetallic compounds and small reprecipitated particles) will show a duplex dimple size. Using this theory of ductile fracture (void nucleation by precipitates) it is possible to derive a mathematical expression relating the total strain to fracture to the volume per cent of inclusions which gives good agreement with experiment.(14) So, the total strain to fracture is a function of the total amount of inclusions while the dimple size is a function of the size and distribution of the inclusions.

Only those inclusions which are not coherent with the matrix or which will fracture easily upon loading are capable of nucleating voids. Two such inclusions have been identified. These are Mg_2Si in aluminum alloys(15) and manganese sulfide in steel(13,16) It is implied that if the



b. 18Cr-12Ni Stainless Steel(8)



d. OFHC Copper(11)



a. AISI 4340 Steel(13)
6,000X



c. Fe-30Ni Alloy(8)

FIGURE 4. ELECTRON MICROFRACTOGRAPHS OF DUCTILE FRACTURES



a. Tensile Fracture⁽⁹⁾

25,900X



b. Shear Fracture⁽⁵⁾

27,000X

FIGURE 5. TENSILE AND SHEAR FRACTURES IN AISI 4340 STEEL, 260 to 280 KSI

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inclusions capable of nucleating voids were removed from a metal, completely ductile material (100 per cent reduction of area at fracture) would result. This is probably not true, however, as void nucleation at grain boundaries and other sites would probably occur.

The picture regarding ductile overload failures in steel and in other body-centered cubic metals and alloys is somewhat clouded by the ductile-to-brittle transition. If conditions (temperature, geometry, etc.) are such that a tough fracture is obtained, the fracture has a dimpled appearance. On the other hand, if conditions are such that the material behaves in a brittle manner, cleavage fractures are obtained, in the absence of grain-boundary weakness. Figure 6 shows a cleavage fracture in iron, broken at 78 K.⁽¹³⁾ This figure exhibits several characteristics of low-temperature brittle fractures in body-centered cubic materials^(2,3):

- (a) Change in orientation of the cleavage plane in crossing grain boundaries (D)
- (b) Indications of the local direction of crack propagation by the river markings on the cleavage facets (arrows)
- (c) Variability of the direction of crack propagation from grain to grain
- (d) Formation of small cleavage "tongues" which are due to the cleavage of deformation twins formed by plastic deformation at the tip of the propagating crack (F).

Turkalo⁽¹⁷⁾ has studied the appearance of cleavage fractures in a plain carbon steel (0.55 C-1.30 Mn) treated to produce a variety of microstructures. A variation in fracture facet size among the various microstructures was observed. In pearlite or upper bainite, the fracture path followed the cleavage plane of the ferrite phase across several pearlite colonies or bainite grains. In lower bainite and tempered martensite the cleavage facets were not as clearly defined but seem to be related to the bainite or martensite platelets. So, it would seem that for the higher temperature decomposition products, the resistance to brittle fracture can be improved by refining the austenite grain size while for the lower temperature, acicular products, the platelet size is the controlling factor, and improvements in toughness may be obtained by reducing the bainite or martensite platelet size. The difference in behavior between the high- and low-temperature products was attributed to the larger number of different orientations of ferrite which may form from a single austenite grain at low transformation temperatures.

The manner in which the transition occurs from a fibrous fracture having a dimpled appearance to a brittle fracture in tempered martensitic steel is a matter of some controversy. The variance may be attributed to the difficulty in interpreting the appearance of low-temperature fractures in tempered martensite. The appearance of these fractures has been termed "quasi-cleavage" because of the absence of clearly defined cleavage facets



6,000X

FIGURE 6. CLEAVAGE FRACTURE IN IRON BROKEN AT 78 K⁽¹³⁾

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and river markings. On one hand it is claimed⁽¹⁸⁾ that the ductile dimples, characteristic of fractures above the transition, increase in diameter and decrease in depth with decrease in temperature until below the transition, the relatively flat, quasi-cleavage appearance is obtained. On the other hand, others claim^(4,19) that the fractures in the transition will consist of discrete areas of dimples and glide-plane decohesion, and quasi-cleavage, the relative amounts varying through the transition range. It is generally agreed, however, that these fractures in steel are too heterogenous for quantitative determination of the stress or temperature, at the time of failure, to be made from the fracture appearance. Determination of such a quantitative relationship would require a statistical analysis of the appearance of a prohibitively large number of replicas and when obtained would apply only to the material and condition for which it was determined.

Components in service are very seldom underdesigned to such an extent that overload could be a cause of failure, and consequently, an initiation site should be looked for. The direction of propagation can usually be determined from the macroscopic chevron markings and the fracture can be traced to an origin which will usually be an area of fatigue fracture, hydrogen-induced fracture, stress-corrosion cracking, or some other type of cracking.

FATIGUE FRACTURES

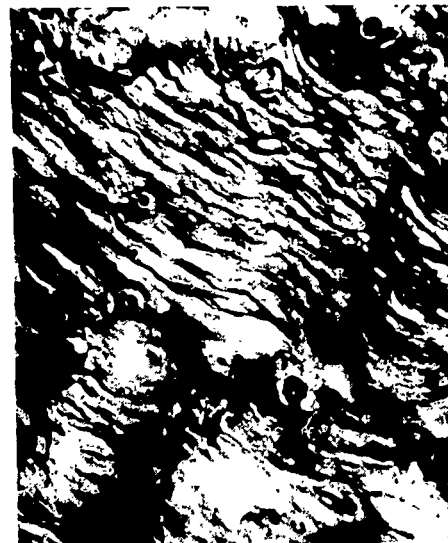
Fatigue fractures are characterized by the appearance of striations normal to the direction of crack propagation on the fracture surface. It has been shown, by the use of programed fatigue-stress cycles, that each stress cycle produces one striation on the fracture surface.^(20,21,22,23) This hypothesis has been further substantiated by comparisons of macroscopic crack growth rates with the microscopic striation spacing^(21,23,24). Typical fatigue fractures in a variety of materials are shown in Figure 7^(8,9,13). Here again, the same general appearance is obtained in essentially all alloy systems. However, as can be seen and as will be discussed in greater detail later, strength level has a profound effect on the appearance of fatigue fractures under the electron microscope.

Forsyth and Ryder proposed a theory for the mechanism of formation of a fatigue striation during one cycle of stress.⁽²²⁾ They assumed that the striations were ridges on the fracture surface that formed in the following manner: the existing crack tip is sharpened during the minimum-stress portion of the cycle. Upon subsequent loading to the high stress, fracture occurs in the region of triaxial stress ahead of the crack which joins the existing crack by thinning of the material between, leaving a ridge on the opposite faces of the fracture. More recently, Laird and Smith⁽²⁵⁾ have shown that the striations are grooves rather than ridges and that the grooves are the direct consequence of the successive rounding, because of plastic flow, and sharpening, at the minimum stress, of the crack tip during each stress cycle.

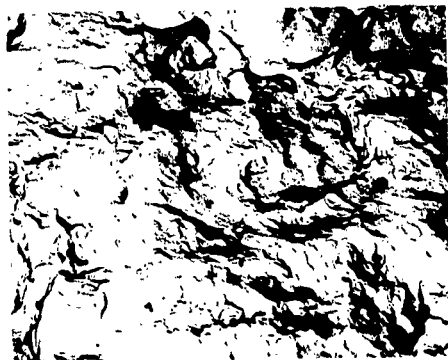
It is to be understood that these theories apply to propagation of existing cracks in fatigue and that these cracks are initiated in the general case, by a slip-governed process at a free surface. Fractographic examination



a. 7178 Aluminum Alloy (13)



c. 18-8 Stainless Steel (8)



d. 4340 Steel, 260 to 280 ksi (9)
13,700X

b. Mild Steel (8)

FIGURE 7. FATIGUE FRACTURES IN VARIOUS MATERIALS

of such initiation areas revealed striations parallel to the direction of propagation(15,25), indicative of a slip process.

As mentioned above, some work has been done correlating striation spacing, stress levels, and distance from the origin in 7000-series aluminum alloys. Reasonable estimates of the cracking stress have been made by measuring the striation spacing as a function of crack length.(24) However, it was necessary to assume a simple stress cycle, and such would not be expected in most service applications. These results indicate that some quantitative work may be possible on fatigue fractures in materials which develop well defined striations, and such correlations would be most valuable in service-failure analysis.

Unfortunately, not all materials display well developed striations on the surface of fatigue fracture. The most notable exception to the general rule is ultrahigh-strength steel. The fractures are relatively flat and featureless, as can be seen in Figure 7d,(9) and if striations appear at all, they are very fine, highly localized, and difficult to detect.(9,16,26) Some investigators feel that the appearance of fatigue fractures in ultrahigh-strength steel may be sufficiently characteristic to enable identification of service fatigue failures, but no quantitative determinations of stress levels would be possible because of the absence of striations. The strength level above which striations do not develop in heat-treated steels has not, as yet, been experimentally determined. Presumably, the absence of striations is due to the inability of the material to flow plastically at the crack tip and thus produce the blunting necessary to striation formation.

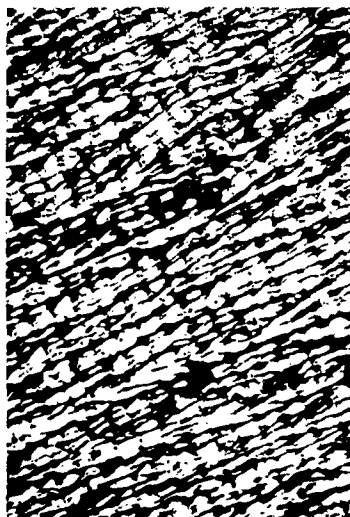
Crussard and his co-workers have shown another case in which striation formation was inhibited. If mild steel was cold worked 15 per cent prior to fatigue testing, striations were not found.(8) Also, striations were not obtained on the fatigue-fracture surfaces in Cu-7Al alloy studied at Ford Scientific Labs.(14) This alloy has a very low stacking-fault energy, causing cross slip to be difficult, so it is implied that cross slip is necessary for striation formation.

The workers at the Royal Aircraft Establishment have observed two types of striations on fatigue fractures in aluminum alloys, which they have termed ductile and brittle(15,27,28). The ductile fatigue fractures (Figure 8a)(27) are the more normal type in which striations having about the same amount of ductile and brittle fracture appear on lozenge-shaped facets. The brittle fatigue fractures appear as striations superimposed on fan-shaped cleavage facets (Figure 8b)(27). The striations on a so called brittle fatigue fracture are wider spaced and more brittle, appearing with cleavage rivers between the striation marks as well as superimposed on them. Ryder also found differences in the appearance of cross sections through the two types of fracture. The ductile-type fatigue fractures had a wavelike appearance with peaks opposite valleys on opposite faces. The striations on the brittle-type fractures appeared to be caused by small cracks off the main crack; they were opposite each other on the two faces of the fracture. Factors which favor brittle-type striations in aluminum alloys are low frequency and corrosive environment at intermediate stress levels. In Armco iron, the presence of hydrogen produced brittle striations in a fatigue fracture.



500X

a. Ductile Fatigue Striations



500X

b. Brittle Fatigue Striations

FIGURE 8. A COMPARISON OF DUCTILE AND BRITTLE FATIGUE STRIATIONS
IN AN Al-7.5Zn-2.5Mg ALLOY LIGHT FRACTOGRAPHS(27)

Intergranular fatigue fractures have been seen by NRL in a titanium alloy(7) and by Ryder(15,21) in an aluminum alloy (Al-7.5Zn-2.5Mg). In these fractures, the fatigue striations were superimposed on grain facets.

In reverse-stress fatigue loadings (such as in a rotating beam test, or in torsion fatigue loading) the striations tend to be smeared and obliterated by the rubbing of the opposite faces against each other. The best developed striations are obtained in tension-tension fatigue panels. No striations are observed in fatigue fractures of ultrahigh-strength steel, even when tension-tension loading is used.

It is generally agreed that the direction of crack propagation in a given grain was perpendicular to the striations in that grain, so the gross direction of propagation could be determined from the average striation direction. Such a determination could be made only for those alloys in which striations develop; it would not be possible for ultrahigh-strength steels.

In attempting to identify fatigue fractures, there are several pitfalls into which a novice could easily fall. Turkalo(17) showed that a cleavage fracture through a pearlite patch has a striated appearance which could be mistaken for fatigue fracture. Plateau(14) reported a similar appearance for ductile fracture in a pearlitic steel. Also, the overstress failures in aluminum casting alloys mentioned later could possibly be mistaken for fatigue failures.

INTERGRANULAR FRACTURE

There are many ways in which intergranular fractures can be produced in metallic materials. Grain-boundary fractures can be produced by failure due to creep at high temperature and low strain rates or by an interaction of applied stress and any of several other conditions such as precipitation or segregation in the grain boundaries, or grain-boundary attack. The problem of intergranular fracture in high-strength steels is of particular importance because hydrogen-induced failures and stress-corrosion failures are both low-ductility grain-boundary fractures. Other conditions which produce or promote intergranular fractures in steel are quench cracking, 500 F embrittlement, temper brittleness, liquid-metal attack and rock-candy fractures in aluminum-killed material.

Figures 9, 10, and 11 are fractographs of a hydrogen-induced brittle fracture, a stress-corrosion crack, and a quench crack, respectively.(13) The marked similarity among the three figures is immediately apparent. All three are definitely intergranular with no visible signs of ductility. A specimen which had been cadmium plated and stressed at a temperature above the melting point of cadmium also showed a similar fracture appearance.(9) The investigators working in the field of electron microfractography generally agree that they cannot distinguish among these types of fracture with any degree of certainty(5,9,13,16,29). However, they are optimistic that some distinguishing features will be found. Perhaps analysis of the "hairlines" which appear on the grain-boundary surfaces will provide such a key. On the other hand, the similarity in fracture appearance may indicate a similarity



7,500X, reduced by 25%

FIGURE 9. HYDROGEN-INDUCED BRITTLE FRACTURE IN AISI 4340 STEEL, ULTIMATE TENSILE STRENGTH, 265 KSI; APPLIED STRESS, 30 KSI; CATHODIC CHARGED

(Courtesy of J. Boyd and J. McCall, Battelle Memorial Institute)

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5,000X

FIGURE 10. STRESS-CORROSION CRACK IN AISI 4340 STEEL(13)

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FIGURE 11. A QUENCH CRACK IN AN AISI 4340 STEEL PART⁽¹³⁾

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in fracture mechanism; especially, hydrogen embrittlement and stress corrosion of high-strength steel may be fundamentally related.⁽²⁹⁾ Until some distinguishing feature is found which will allow differentiation among the various types of intergranular fracture, service-failure analysis will require supporting information such as the processing, service conditions, and general appearance of the failed part.

Low⁽³⁰⁾ in discussing cleavage fractures mentioned that there was no characteristic difference between fractographs of the intergranular fracture surface of temper brittled and unembrittled steels, although the Charpy transition temperature was shifted upwards 200 C. Intergranular brittle fractures in Charpy V-notched and center-notched sheet tensile specimens of AISI 4340 and experimental steels after tempering at 500 to 600 F have also been reported.⁽³¹⁾ This may be related to 500 F embrittlement, which has long been recognized in tempered alloy steels.

Another group of intergranular fractures are those which are induced by the interactions of a stress field and intergranular precipitates or grain-boundary precipitates. Under the electron microscope these fractures reveal large areas covered by a precipitate with the remainder of the fracture consisting of grain surfaces covered with ductile dimples. Evidently, the grain-boundary-precipitate films either cleave or separate from the adjacent grains, and the voids thus produced nucleate ductile failure of the remaining grain-boundary area. The low ductility or low energy absorption of these fractures is due to the large amount of precipitate concentrated on the grain surfaces rather than to any weakness of the matrix material. Since the grain-boundary precipitates nucleate dimples on the remainder of the surface, these fractures might be termed intergranular ductile fractures.

Two notable examples of this type of fracture might be mentioned. These are the intergranular fracture of steels containing appreciable amounts of aluminum and nitrogen (rock-candy fracture)⁽³⁾ and the fracture of austenitic chromium-nickel steel due to grain-boundary carbide precipitation.⁽³²⁾ Figure 12⁽³⁾ is a fractograph of an extraction replica of the fracture surface of an embrittled aluminum-killed steel casting showing the thin grain-boundary precipitate, most of which was dendritic in form and which was determined by electron diffraction to be AlN. The study performed on the stainless steel was quite similar. Precipitates on the grain surfaces were usually in the form of flat dendrites covering perhaps 50 per cent of the surface, and were identified as chromium carbides. These studies showed that thin discontinuous grain-boundary films, often difficult to detect by normal metallographic techniques, may be readily studied and even analyzed by using electron microfractography.

A third type of intergranular separation which occurs in metallic materials is the fracture at elevated temperatures under sustained loads. Under conditions of relatively high temperature and low stresses (above the so-called equicohesive temperature) creep fractures occur by the formation and growth of intergranular fissures. It has been shown that on the final fracture in both iron and in austenitic steels the surfaces of the fissures are covered with striations which are oxidation figures produced by absorption of oxygen on the free surface (thermal etching).⁽³³⁾ Oxygen, either dissolved in the metal or diffusing from the surrounding atmosphere, is



3,500X

FIGURE 12. INTERGRANULAR FRACTURE IN A STEEL CASTING CAUSED BY DENDRITIC AlN PRECIPITATES IN THE GRAIN BOUNDARIES(3)



FIGURE 13. ELECTRON MICROFRACTOGRAPH OF AN
INTERGRANULAR FRACTURE IN AN ALUMINUM-
SILICON-COPPER ALLOY⁽³⁴⁾

absorbed on the internal surfaces of the cavities, where it produces the observed geometric patterns.

A similar striated appearance has been observed on the surfaces of intergranular fracture in nickel(14) and cast aluminum alloys(16,34,35). Figure 13(34) is an electron microfractograph of such a fracture in an aluminum-silicon-copper alloy and shows the patterns of fine lines on the fracture surfaces. The manner in which the striations form is a matter of controversy among the investigators. Investigators at Boeing feel that they represent fracture through the many intermetallic compounds found in the grain boundaries of the cast aluminum alloys. Ryder believes the striations are produced by the interaction of the advancing crack front with elastic stress waves in the material. On the other hand Plateau attributes the striated appearance to the segregation of oxygen to the grain boundaries, where it produces an effect similar to that described above for the surface of fissures formed during creep. In this case, the surfaces to be considered are the grain surfaces themselves rather than the surface of a fissure. This difference of opinion should be resolved by future research.

APPLICATION TO SERVICE FAILURE ANALYSIS

It is readily apparent from the figures attached to this memorandum, that sufficient distinguishing features exist, among the various types of fractures, to enable electron microfractography to be used as a tool in the analysis of service failures. It is possible to distinguish overload failures from fatigue failures from brittle intergranular failures and so on. However, at present, it is not possible to make fine distinctions within any one category of fracture as to the stress, temperature, or other factors existing at the time of failure. Future research may enable such evaluations to be made.

Especially in the case of ultrahigh-strength steel care is needed in interpreting service failures by fractography. This is due, in part, to the absence of distinct fatigue striations and also to the hard-to-interpret, quasi-cleavage appearance of fractures below the transition in these materials. Fractography should be used as a tool to support existing techniques and should not be expected to replace them. In most cases, information regarding the material and its processing as well as the service conditions and environment will be needed before a definitive answer as to the cause of a particular failure can be given. For example, Figure 10(13) is the fractograph of the fracture origin of a service failure which visually appeared to have been caused by fatigue, but fractography showed the fracture to be intergranular; a knowledge of the part and its service environment indicated the cause to be stress corrosion.

So, although limitations do exist, electron fractography is now a useful technique for service-failure analysis, and future research should steadily increase its usefulness and applicability.

POSSIBLE AREAS FOR FUTURE RESEARCH

In the course of surveying the work which has been done on electron microfractography, it became evident that there are several areas where future effort could be expended to increase the knowledge of fracture mechanisms and to increase the usefulness of the tool for service-failure analysis. First of all, efforts should be made to reconcile the differences in interpretation which exist among the various investigators in the field. Also, the presently used replication techniques are those which were developed for electron microscopy of metallographic specimens. Technique should be developed specifically for fractography which would combine the advantages of the direct carbon method (better detail and fewer artifacts) and the plastic-carbon method (the specimen preserved for further studies). Systematic studies of fatigue fracture appearance as a function of strength level, stress level, and composition are needed. In those alloys where striations are well developed, further efforts should be made to obtain quantitative relationships among striation spacing, stress levels, and other material and loading variables. Additional systematic studies are needed to enable distinctions to be made between the various types of intergranular fracture. Since hydrogen-induced delayed brittle failure and stress-corrosion cracking in high-strength steels are extremely critical current problems, considerable effort in these areas is certainly justified. Considerably more work is needed to establish the relationships between fracture appearance and microstructural features. Such studies would involve the preparation of both plastic-carbon and carbon extraction replicas of fracture surfaces and metallographic studies of nickel-plated cross sections through fractures to ascertain the relationship among fracture path, microstructure, and inclusion patterns. Information obtained from such studies may indicate ways in which controlling the microstructure and inclusion distribution would improve the properties of various metallic materials.

Electron microfractography is certainly a useful and promising metallurgical tool and is expected to see an ever-increasing field of application.

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